भारतीय मानक
Indian Standard

IS 1448 [P : 128] : 2018

पेट्रोलियम और उसके उत्पादों के लिए परीक्षण के तरीके

[पी : 128] कलसिनेड पेट्रोलियम कोक में निकल का निर्धारण

(पहला पुनरीक्षण)

Methods of Test for Petroleum and Its Products

[P : 128] Determination of Nickel in Calcined Petroleum Coke

(First Revision)

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Methods of Sampling and Test for Petroleum, Petroleum Products, Gaseous Fuels and Lubricants Sectional Committee, PCD 1

FOREWORD

This Indian Standard [P : 128] (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Methods of Sampling and Test for Petroleum, Petroleum Products, Gaseous Fuels and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The presence and concentration of nickel elements in a petroleum coke are major factors in the suitability of the coke for various uses. Measuring the amounts of nickel elements in the coke sample for use by the purchaser and the seller in the commercial transfer of petroleum coke to determine whether the lot of coke meets the specifications of the purchasing party. This test method provides measuring commercially important metallic elements in coke samples.

This standard was first published in 1982. In this revision, provision for using AAS and ICP spectrophotometer is included.

In reporting the results of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 128] DETERMINATION OF NICKEL IN CALCINED PETROLEUM COKE

(First Revision)

1 SCOPE

This standard describes an analytical procedure for the determination of nickel in calcined petroleum coke.

2 APPARATUS

2.1 Volumetric Flasks — 1 000 ml, 500 ml, 250 ml, and 100 ml capacities.

2.2 Pipettes — 25 ml, 10 ml and 1.0 ml capacities.

2.3 Measuring Cylinders —100 ml and 10 ml capacities.

2.4 Porcelain Dish — 250 ml capacity.

2.5 Muffle Furnace — Capable of controlling the temperature between 600 and 1 000°C with regulator.

2.6 Spectrophotometer — UV visible or AAS or ICP.

3 REAGENTS

3.1 Dimethylglyoxime Solution (1 percent) — Dissolve 1 g of the above reagent in 100 ml of ethyl alcohol.

3.2 Citric Acid (10 percent Solution) — Dissolve 10 g of citric acid ($C_6H_8O_7.H_2O$) in 100 ml of distilled water.

3.3 Potassium Persulphate (2 percent Solution) — Dissolve 2 g of potassium persulphate ($K_2S_2O_8$) in 100 ml of distilled water (freshly prepared).

3.4 Sodium Hydroxide Solution (2M) — Dissolve 8 g of NaOH pellets in 50 ml of water, cool and make up to 100 ml with distilled water.

3.5 Nitric Acid Solution (8N) — Dilute 500 ml of HNO_3 (sp gr 1.42) to 1 litre with distilled water.

3.6 Hydrochloride Acid Solution (1 N)

3.7 Sodium Carbonate, Anhydrous

3.8 Boric Acid

4 STANDARD NICKEL SOLUTION

4.1 Preparation of Standard Nickel Solution (1 ml = 0.1 mg Ni) — Dissolve 0.1 g of pure nickel metal (99.99 percent) in 10 ml water and 5 ml nitric acid in a beaker. When the dissolution is complete, boil the solution to remove the lower oxides of the nitrogen. Cool and make up to 1 000 ml in volumetric flask with distilled water and mix. Transfer the contents to 500 ml polyethylene bottle tightly covered.

4.2 Preparation of Standard Nickel Solution (1 ml = 0.005 mg Ni) — Dilute 25 ml of the stock solution (*see* **4.1**) in a 500 ml volumetric flask and make up to the mark with distilled water and mix. Transfer the contents to 500-ml polyethylene bottle having tight fitting stopper.

4.3 Preparation of Calibration Curve

4.3.1 Transfer 2, 4, 6, 8 and 10 ml aliquots of standard nickel solution (1 ml - 0.005 mg Ni) (*see* **4.2**) into 100 ml beakers. Add 5 ml citric acid to each beaker and neutralize each solution using litmus paper by ammonia (1: 1) and add 5 ml excess. Transfer the solution to a clean separating funnel and dilute to 50-70 ml with distilled water. Introduce 10 ml Dimethylglyoxime solution and mix. Extract with three 5 ml portions of chloroform ($CHCl_3$) shaking vigorously (1-2 min) each time and combine the chloroform layers in a clean separatory funnel. Shake the combined chloroform extract with 20 ml of 0.5 M ammonia and draw off the chloroform layer into another clean separatory funnel. Shake the ammonical aqueous phase with 5 ml of chloroform. Separate the chloroform layer and add this to the main portion which contains nickel.

4.3.2 Shake well (1-2 min) the combined chloroform extract with two 5 ml portions of IN HCl and transfer both acid layers into a 100 ml volumetric flask, dilute each flask to about 50 ml with distilled water, add 1 ml of citric acid, 3 ml of potassium persulphate solution, 15 ml 2M sodium hydroxide solution and 1 ml of dimethylglyoxime solution and mix. Heat to 60°C in water bath and keep it at 60-70°C for 5 min, cool and dilute to 100 ml with distilled water and mix.

4.3.3 Measure absorbance after 10 min at a wavelength of 465 nm in 1 cm cell on a Spectrophotometer. Run a blank using the same quantity of reagents except the nickel solution.

4.3.4 Plot the photometric readings of calibration solution against milligrams of nickel per 100 ml of solution.

5 PROCEDURE

5.1 Ash 50 g of calcined petroleum coke sample after passing through 250 micron sieve in a porcelain dish at 950°C. Transfer the whole ash using camel hair brush to a platinum dish.

5.2 Add 6 g of anhydrous sodium carbonate (Na_2CO_3) and 2.5 g of boric acid, mix thoroughly with glass rod, clean the glass rod with camel hair brush. Cover the dish with lid and heat slowly with burner for 10 min, avoid spurting, and finally heat the dish in a muffle furnace at temperature of 950 ± 50°C for 30 min. Take out the dish and cool.

5.3 Extract the mass of the dish with 43 ml of 8N HNO_3, boil the solution for few minutes, cool and transfer into a 250 ml volumetric flask, make up to the mark with distilled water and mix.

5.4 Take 10 ml aliquot from above solution (*see* **5.3**) into a 250 ml beaker, add 5 ml of citric acid and proceed as in **4.3**.

5.5 Run a blank simultaneously through the entire procedure using all reagents and dilution exactly the same way excluding the sample.

5.6 Measure the absorbance after 10 min at 465 nm for the blank and sample. Deduct the reagent blank and calculate net milligrams of nickel (Ni) in the aliquot.

6 CALCULATION

$$\text{Nickel (Ni)} = \frac{\text{Net mg of Ni in aliquot} \times \text{Aliquot factor percent by mass} \times 100}{1\,000 \times \text{mass of sample}}$$

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